SLOVENSKI STANDARD
SIST EN 196-11:2019

01-februar-2019

Metode preskušanja cementa - 11. del: Toplota hidratacije - Izotermna kondukcijska kalorimetrija (ICC)

Methods of testing cement - Part 11: Heat of hydration - Isothermal Conduction Calorimetry method

Prüfverfahren für Zement - Teil 11: Bestimmung der Hydratationswärme von Zement durch isotherme Wärmeflusskalorimetrie

Méthodes d’essais des ciments - Partie 11: Chaleur d’hydratation - Méthode par calorimétrie à conduction isotherme

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ICS:

91.100.10	Cement. Mavec. Apno. Malta	Cement. Gypsum. Lime. Mortar

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EUROPEAN STANDARD

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (EN 196-11:2018) has been prepared by Technical Committee CEN/TC 51 "Cement and building limes", the secretariat of which is held by NBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2019, and conflicting national standards shall be withdrawn at the latest by September 2020.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

EN 196 consists of the following parts, under the general title *Methods of testing cement:*

— *Part 1: Determination of strength;*

— *Part 2: Chemical analysis of cement;*

— *Part 3: Determination of setting times and soundness;*

— *Part 4: Quantitative determination of constituents* (CEN/TR 196-4);

— *Part 5: Pozzolanicity test for pozzolanic cement;*

— *Part 6: Determination of fineness;*

— *Part 7: Methods of taking and preparing samples of cement;*

— *Part 8: Heat of hydration - Solution method;*

— *Part 9: Heat of hydration - Semi-adiabatic method;*

— *Part 10: Determination of the water-soluble chromium (VI) content of cement.*

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This document specifies the apparatus and procedure for determining the heat of hydration of cements and other hydraulic binders at different test ages by isothermal conduction calorimetry.

This test procedure is intended for measuring the heat of hydration of cement up to 7 days in order to obtain correspondence between Isothermal Conduction Calorimetry (ICC) and EN 196-8 and EN 196-9. Nevertheless this test duration may be critical for some apparatus, even if they can work properly at shorter test ages.

Contrary to EN 196-8 this method gives the heat of hydration continuously over the time. Additionally, the heat flow versus time is given.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
isothermal conduction calorimeter
apparatus able to measure the heat flow generated by a sample kept at constant temperature

Note 1 to entry: The constant temperature condition is achieved by maintaining the sample in thermal contact with a heat sink.

3.2
output of calorimeter
electric signal from the calorimeter expressed in V

3.3
thermal power
heat rate produced by the sample during the test

Note 1 to entry: It is commonly expressed, with reference to the unit mass of cement, in W/g or J/(s×g).

3.4
heat
time integral of the thermal power and expressed in J/g

3.5
calibration coefficient
ε
ratio between the thermal power produced in the calorimeter and the output of the calorimeter

Note 1 to entry: Expressed in W/V.

3.6
baseline output
BO
output of the calorimeter when there is an inert sample in the testing and reference cell, both with the same thermal capacity

Note 1 to entry: Expressed in V.

3.7
baseline drift
BD
representation of the slope of the linear regression of the baseline output versus time measured over a specified period

Note 1 to entry: It is expressed in W/g per time period, with reference to the unit mass of cement[1]. The baseline drift measured in V per time period and unit mass of cement, is converted in unit W per time period and unit mass of cement by the calibration coefficient.

3.8
baseline noise
BN
representation of the standard deviation of the regression of the Baseline output versus time measured over a specified period

Note 1 to entry: It is expressed in W/g, with reference to the unit mass of cement.

3.9
testing cell
testing ampoule
measuring cell specifically dedicated to the sample under test

Note 1 to entry: It is the sample that generates heat.

3.10
reference cell
reference ampoule
measuring cell specifically dedicated to an inert sample

Note 1 to entry: It is a sample that doesn't generate heat and it is used to reduce the *BD* and *BN.*

3.11
ampoule
container into which the sample is placed for a measurement

3.12
ampoule holder
holder into which the ampoule is placed for a measurement

Note 1 to entry: The ampoule holder conducts the heat from the sample in the ampoule to the heat flow sensor.

[1] The mass of cement to be used is the mass of the sample that will be used for the measurement.

3.13
time constant
τ
order of magnitude of the time needed to reach the new thermal equilibrium

Note 1 to entry: It is a measure of the thermal inertia of the test cell and it is expressed in s.

3.14
detection limit
DL
minimum value of thermal power that an apparatus is able to detect

Note 1 to entry: This value is an estimation of the quality of the measurement that is dependent on the whole measuring chain and not only on the design of the instrument.

4 Apparatus

4.1 General

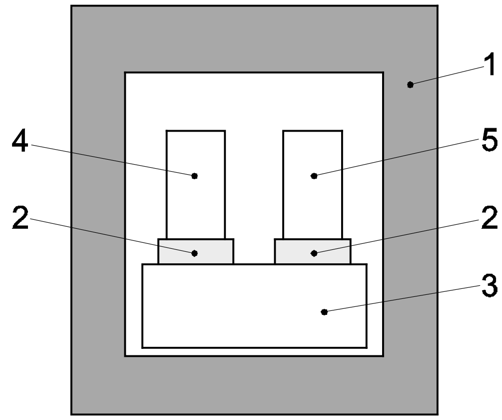
This part gives general requirements related to the relevant properties and design of a calorimeter. Although the design of individual calorimeters from different manufacturers may vary, it should meet the specifications as described below.

4.2 Principle

An isothermal heat conduction calorimeter is called “isothermal” as the temperature changes in it (if the instrument and the method are well designed) are so low so that the results are – from a practical point of view – the same as if the measurement had been made at perfectly isothermal conditions. The term is convenient to use here as most other cement calorimeters are (semi-) adiabatic, but it does not imply that the measurements are made under perfectly isothermal conditions.

An isothermal heat conduction calorimeter has to consist of a constant-temperature heat sink to which at least two heat-flow sensors and sample holders (calorimetric cells) are attached in a manner resulting in good thermal conductivity. One cell contains the sample of interest and the other one, the reference cell, contains a blank sample that doesn’t evolve heat. If the calorimeter contains more than two cells, at least one has to act as reference cell. Often the conduction calorimeters with more than two cells have own reference cells for each of the sample cells. The cells, including the holder, shall have the same heat capacity on the reference side and on the sample side. This may be realized by adopting the same design and materials on both sides. If an inert sample with similar heat capacity as the sample is charged into the reference ampoule, noise and drift will be substantially decreased (balancing).

The heat released by the hydrating cement sample, flows across the sensor into the heat sink. The voltage output from a measurement is the difference between the voltages from the test cell and the reference cell. The heat-flow sensor senses the small temperature gradient that develops across the device. However, the heat is removed from the hydrating sample fast enough that, for practical purposes, the sample remains at a constant temperature (isothermal).



Key

1 thermostat

2 heat flow sensors

3 heat sink

4 sample

5 reference

Figure 1 — Schematic drawing of a heat conduction calorimeter

4.3 Thermostat

Although the driving force of heat flow is a temperature gradient, the overall temperature in the calorimeter (heat sink) during test should be essentially constant (isothermal). For this purpose the calorimeter should be equipped with a thermostat. The temperature instability of the thermostat should not exceed 0,2°C. The working temperature of the apparatus should be at (20 ± 0,2) °C.

4.4 Calorimeter technical parameters

The technical parameter of the calorimeters that have to be specified to perform the heat of hydration determinations and to ensure an acceptable robustness of the results are the following:

— Detection limit (*DL*);

— Baseline noise (*BN*);

— Baseline drift (*BD*).

The calorimeter has to meet the following specifications:

— *DL*: < 2,8 µW/g;

— *BN*: < 10,5 µW/g;

— *BD*: < 5,5 µW/g per week.

NOTE The above specifications have been defined on the basis of the technical specifications of the apparatus currently used for calorimetric measurements on hydraulic binders. For the time being there are no experimental data correlating these specifications with the trueness and precision (repeatability and reproducibility) of the heat of hydration results.